Combined Synchrotron X-ray Diffraction Studies to Synthetic Gallogermanate Cancrinite: 3. *In Situ* Synchrotron X-ray Powder Diffraction Studies

Y. Lee, J.B. Parise (SUNY, Stony Brook) and J.C. Hanson (BNL, Chemistry)

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Beamline(s): **X7B**

Time-resolved synchrotron X-ray powder diffraction patterns were obtained during the 5 hours of the *in situ* heating of GaGe-CAN between RT and 800°C [1]. One set of peaks, which could be indexed as cancrinite, persisted up to 550°C. During this period, a continuous increase in the hexagonal lattice parameters was observed as can be seen from the shifts of the set of peaks to lower 2θ values. According to the thermogravimetry result, this period also corresponds to the dehydration of the $Ge(OH)_6$-octahedra. A second phase appeared at 550°C, where the dehydration of the $Ge(OH)_6$-octahedra was complete. This new phase was indexed on an orthorhombic cell and was identified as a GaGe-analogue of nepheline-hydrate I (NHI), a tetrahedral framework structure with intersecting 6-ring and 8-ring channel system (JCPDS data file 75-1740) [2]. The unit cell parameters of GaGe-NHI were refined to *a* = 8.348(1), *b* = 15.865(2), *c* = 5.351(1) Å, using a set of data integrated to represent a 10 min portion between 550 and 560°C. After the formation of GaGe-NHI at 550°C, a third phase started to grow, while a fourth phase appeared from 600°C. These two unknown phases disappeared at 790°C, from which sets of broadened peaks were observed with increased background until the final temperature of 800°C.

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References: [1] Y. Lee, J.B. Parise, A. Tripathi, S.J. Kim, and T. Vogt. (2000) Microporous Mesoporous Mater., 39, 445. [2] S. Hansen, L. Falth, Zeolites 2 (1982) 162.

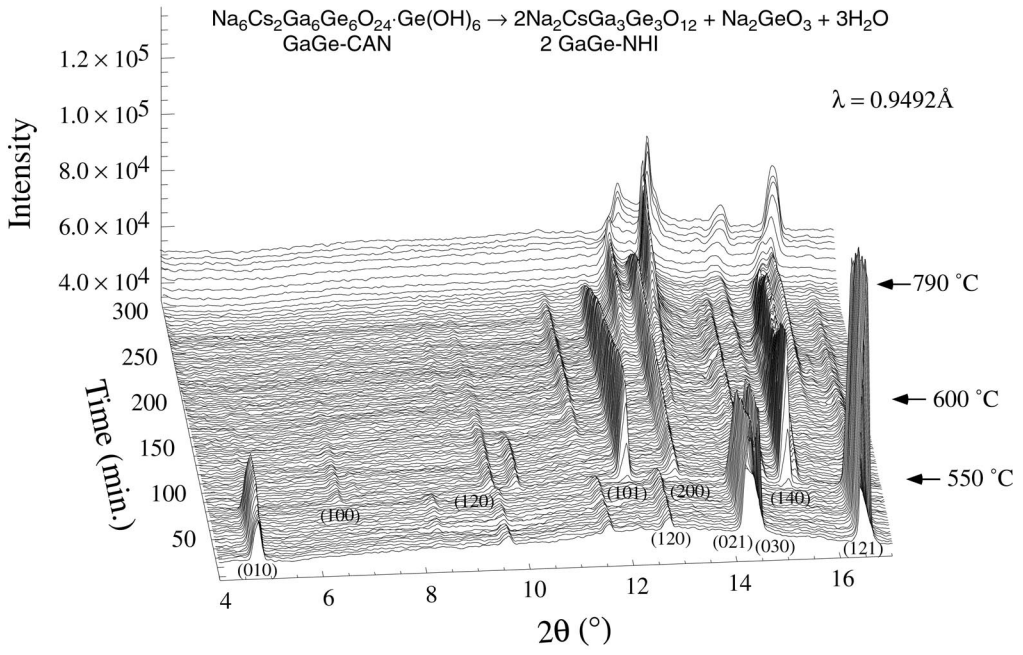

Figure. Plot of the synchrotron X-ray powder diffraction profiles as a function of time during the 5 hours of the *in-situ* heating of GaGe-CAN. Some of the reflections from the GaGe-CAN and GaGe-NHI phases are marked with their indices. The patterns were obtained by integrating the imaging plate vertically with an integration width of 3 mm, about the size of the slit.